Review Article

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Green synthesis of nanoparticles: A review

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Abstract

Green synthesis is more beneficial than traditional chemical synthesis because it costs less, decreases pollution, and improves environmental and human health safety. In this review, current developments in the green synthesis of nanoparticles In particular, the metal nanoparticles are synthesized by top-down and bottom-up approaches through various techniques like physical, chemical, and biological methods. Their characterization is very vital and the confirmation of nanoparticle traits is done by various instrumentation analyses such as UV–Vis spectrophotometry (UV–Vis), Fourier transform infrared spectroscopy (FT-IR), scanning electron microscope (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD), atomic force microscopy (AFM), were evaluated. Major findings reveal the complexity in geographical and seasonal distributions of plants and their compositions that green synthesis is limited by time and place of production as well as issues with low purity and poor yield. However, considering current environmental problems and pollution associated with chemical synthesis, green synthesis offers alternative development prospects and potential applications.

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INTRODUCTION

The term nanometallic materials refers to metals and alloys that form nanocrystalline grains with particle sizes ranging from 5 to 100 nm [1]. In comparison to their non-nano or bulk counterparts, metals at the nanoscale have a higher surface area. Numerous varieties of nanoscale metals are used extensively in biology, medicine, and engineering [2] for example Au nanoparticles

(Au NPs) have biological application for enzyme regulation and antimicrobial and muscle relaxant activities [3]. Ag NPs inhibit growth and activities of both gram-positive and gram-negative bacteria [4]. Fe NPs also inhibit bacterial growth and are effective in cleaning up matrices contaminated with organic matter, metal and non metal ions and dyes [5]. Pd NPs are also used in various applications in dye degradation, antimicrobial activity, and catalysis [6]. Nowadays, more studies are being done on the green, physical, and chemical synthesis of nanoscale metals [7]. Physical and chemical methods are gradually being replaced by green synthesis methods because of issues related to consumption of large amount of energy [8], release of toxic and harmful chemicals and use of complex equipment and synthesis conditions. Currently, green synthesis mostly uses microorganisms (fungi, bacteria, and algae), or extracts from leaves [9], flowers, roots, peelings, fruits, and seeds of different plants.

Advantages:

Green synthesis has many advantages compared to chemical and physical methods:

- It is non-toxic
- Pollution-free
- Environmentally friendly
- Economical
- More sustainable.

Green synthesis of nanoparticles:

Gold NPs: Typically, plant extracts or microorganisms are used as reducing agents to reduce gold ions during the green procedure of Au NPs. In order to obtain the extracts, the ground plants are often soaked in solvents (such as water or ethanol) under favourable environmental circumstances (various green substances have different ideal conditions). The extracts are then mixed with a solution containing gold ions, and Au NPs are produced when the solution turns red [10].Using this method chloroauric acid was converted to gold nanoparticles using extracts from the leaves of Cassia auriculate and Cinnamomum zelyanicum, respectively. The most common shapes of green synthesized Au NPs are spheres, and some are triangles and hexagons.

For example, using aqueous extract of Garcinia mangostana fruit peels. In a conical flask, 20ml of the peel extract was reacted with 10M of tetrachloroaurate at room temperature under static conditions. The colour change of the reaction was observed and the time taken for the changes was noted. The solution colour changes immediately form pale brownish to purple colour indicating the formation of [Au/G. Mangostana]. The Au-NPs nanoparticles emulsion obtained was kept at 4℃ [11].

Silver NPs: The common green synthesis for Ag NPs involves mixing of silver nitrate solution with reducing substances extracted from plants. The generation of Ag NPs is indicated by the solution turning to brownish colour [12]. The green synthesized Ag NPs vary in shape and size and the most common forms are spherical, triangular, and hexagonal. There are several factors affecting the green synthesis of Ag NPs: plant extract, pH, and temperature. Regarding the applications of Ag NPs, these NPs are also powerful catalysts and microbial growth inhibitors. Researchers discovered through the disk diffusion experiment that Ag NPs had a good inhibitory impact on both gram-positive and gram-negative bacteria, which Au NPs were unable to accomplish. Additionally Ag NPs can function as photocatalysts in electronic and medical equipment.

For example, using extract of Aloe barbadensis, 0.1M silver nitrate solution was prepared in distilled water. 10ml, 15ml and 20ml of the silver nitrate solution was taken in a glass beaker and kept in magnetic stirrer for 15min at 65℃. 1ml of plant extract was added drop wise in different volume of $AgNO_3$ solution with continuous stirring. The mixture was kept on magnetic stirrer for 15min to observe colour change to reddish brown. Change in colour indicates synthesis of NPs.

Palladium NPs: Palladium is a precious high-density metal. It is commonly used in medical diagnostics and as catalyst and biosensor. For example, using Glycine max (soybean) plant extract. Take 10ml of plant and add 100ml $PdCl_2$ solution. Put this mixture on magnetic stirrer with hot plate at 60℃ with constant stirring for 30min, at the starting point of the reaction. We observe the colour change light orange to dark brown colour obtained [13].

Copper NPs: Cu belongs to the group of light transition metals, so Cu NPs usually cannot just be obtained directly from simple copper salts. Its need capping agents such as surfactants to control the size of particles.

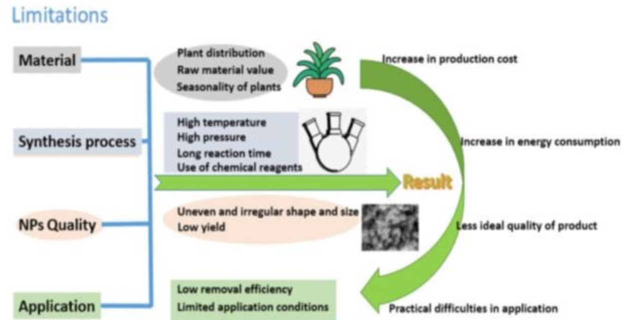
For example, using Fortunella margarita leaves extract, 5gm of leaves of Fortunella margarita were washed and crushed finely into a thin paste by using pestle mortar. The paste was diluted with distilled water up to 100ml and was kept at room temperature. After 1hr, the mixture was filtered to obtain the phytochemicals (reducing agents) for the reaction. The filtrate was then mixed with 1M of copper sulphate solution was kept in a water bath at 70℃ for 30min. Synthesized CuNPs were then collected and washed thrice by using a centrifuge at 12,000 rpm for 1hr at 30℃. After washing, copper nanoparticles were lyophilized/dried to obtain the powdered copper nanoparticles [14].

Iron NPs: Many nanoparticles for green synthesis of Fe NPs have been studied. Extracts from mango leaves, eucalyptus leaves, and grape see, pear tree leaves, vine leaves have been used for the green synthesis of iron nanoparticles that exhibited strong antioxidant capacity. The synthesis of nanoscale zero-valent iron is more complex than that of other nanoparticles. Grape seed extracts worked effectively as a stabilizing agent and prevented the oxidation and agglomeration of NZVI because of polyphenol and proanthocyanidins that eliminated reactive oxygen species including peroxyl and hydroxyl radicals [15]. The polyphenols also has the ability to promote the stability of the nanoparticle by serving as capping agents.

For example, using Neem (Azhadirachta indica) leaves extract, In FeNPs synthesis, the plant extract is mixed with 0.001M hydrated ferric chloride solution at the temperature range 50-60℃. The change in colour from brown to black indicates the formation of iron nanoparticles [16].

Limitations:

- Green synthesis of nanoscale metals has great potential, however, it suffers setbacks from material selection, synthesis conditions, and product quality control and application.
- These parameters present challenges towards adopting industrial production and large scale application of green synthesized nanoscale metals.



Materials:

There are many plant materials for green synthesis of NPs, and several researchers have studied plants that are available locally and in abundant supply. It provide the possibility to make full use of local plants, but it is difficult to achieve large scale production of nanoscale metals. For example, Pd NPs was synthesized from Lithodorahispidula, green synthesis of Au NPs, NZVI, iron oxide NPs, and Cu NPs are also useful for local plants. Fenugreek used in the synthesis of Au NPs. Oolong tea, a unique variety of tea in china that was used in the production of nanometer zero-valent iron. Others, such as psoralen, are also used for the synthesis of iron oxide NPs, but the materials are mainly distributed in India, Myanmar, and Sri Lanka.

Time constraints also hinder the use of raw materials in actual production. In addition, some raw materials belong to secondary products that need further processing, which add complexity and cost technology.

Synthesis process:

The main concerns in the synthesis process are excessive energy consumption, long reaction time, and use of other industrial chemical reagents. Ferulapersica's root and leaf extracts had been used to synthesize Ag NPs at 600℃ for three hours [17], and guava fruit extract was used to synthesize Cu NPs in 800℃ water bath [18], compared with the chemical synthesis method, CuO NPs can be obtained by ultrasonic stirring at 80℃ for 2hr. Therefore, the temperature required for some green synthesis processes is extremely high and the synthesis time is quite long, which require intensive energy that may have an adverse impact on the environment.

NPs quality:

The size and shape of nanoparticles synthesized by different extracts are highly variable, and the properties determined are insufficient. Current reports show big differences in particle size, which makes green technology not suitable for wide scale production or controlling particle size during production. The particle size of NZVI NPs synthesized from grape seeds varied from 63 to 381 nm, while the size of Ag NPs synthesized from Nigella arvensis leaf ranged from 5 to 100 nm [19]. However, the nanoparticles synthesized by physical method can exhibit high purity and uniform particle size distribution. This phenomenon reflects the low conversion rate and low utilization rate of metal ions, which means only small number of nanoparticles can be synthesized from a large concentration of metal ions, thus the economic benefit is low.

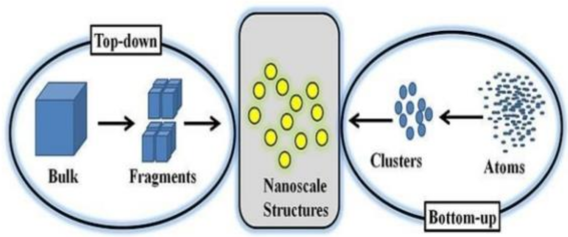
Applications:

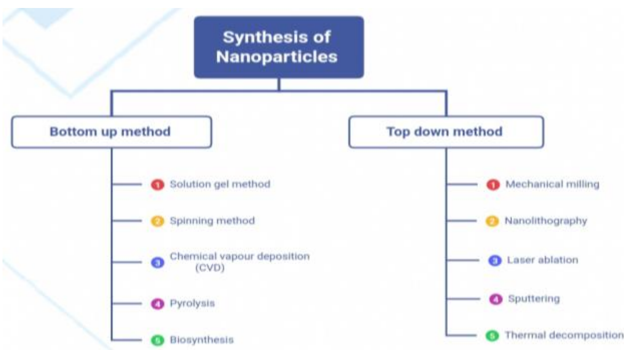
Metal nanoparticles have large surface area, strong absorption, and high reducibility that they can be used in removing pollutants. However, not all metal nanoparticles synthesized through green synthesis can remove environmental pollutants. The COD and BOD removal efficiencies of NZVI synthesized by Spinacia oleracea (spinach) were not high: 73.82% and 60.31% respectively [20], after 15days. These low removal rates of NZVIs cause concerns especially on inefficient use or waste of iron. Green synthesized nanoscale metals exhibit low efficiency in removing mixed toxic metals. For practical applications, heavy metals and other pollutants in sewage are mixed and rarely a waste contains only one heavy metal.

Application of green synthesized nanoscale materials on in vitro cells is limited. The study showed the dose-dependent inhibition of iron oxide nanoparticles on human breast cancer cells. Renal tumor cells can also be inhibited at low concentration [21]. These studies have been carried out in vitro; the effects of green nanoparticles in vivo are unknown.

General method of synthesis of Nanoparticles:

The numerous technique used to create the nanoparticles can be divided into two categories: top-down and bottom-up techniques.



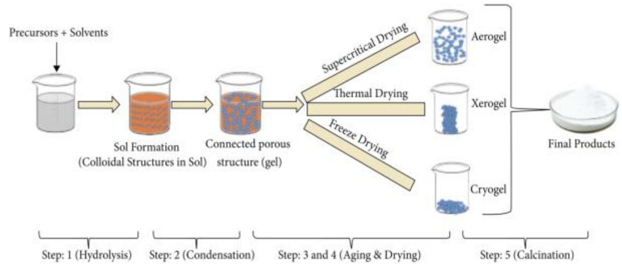


I) Bottom up method:

The construction of material from atoms to clusters to nanoparticles is known as the bottom up or constructive method. The most popular bottom up techniques for producing nanoparticles includes solution gel, spinning, chemical vapour deposition, pyrolysis, and biosynthesis.

Solution-gel method:

Steps involved in solution-gel method



The solution is a suspension of particles in a liquid phase called a colloidal solution. The gel is an insoluble solid macromolecule. Due to itssimplicity and the ease with which the majority of nanoparticles may be created, solution gel is the most used bottom upapproach. It is a wet chemical method that uses a chemical solution as a precursor for an integrated system of discrete particles.

Commonly utilized precursors in the solution gel process are metal oxides and chloride [22]. . A system with a liquid and solid phase is created after the precursor is distributed in a host liquid via shaking, stirring, or sonication. Different techniques including sedimentation, filtering, centrifugation, are used in phase separation to recover the nanoparticles, and drying is used to further remove the moisture [23].

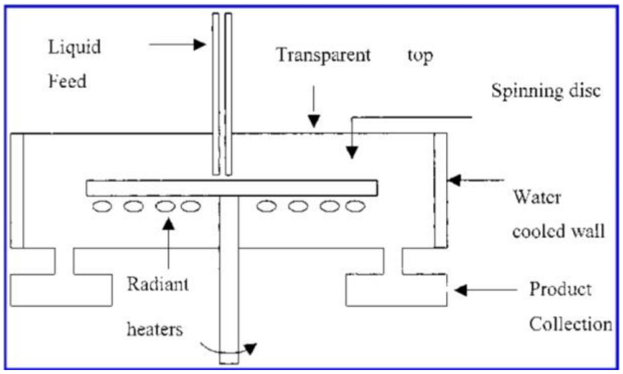
Advantages:

- Synthesis of metal oxide nanoparticles.
- Cost-effectiveness and high chemical product composition control are two benefits of the sol-gel method.
- Ability to produce thin layers of amorphous materials using this approach,
- Synthesis products with high purity.

Disadvantages:

- The disadvantage of sol gel method is the high cost of alkoxide precursors.

Spinning Method:

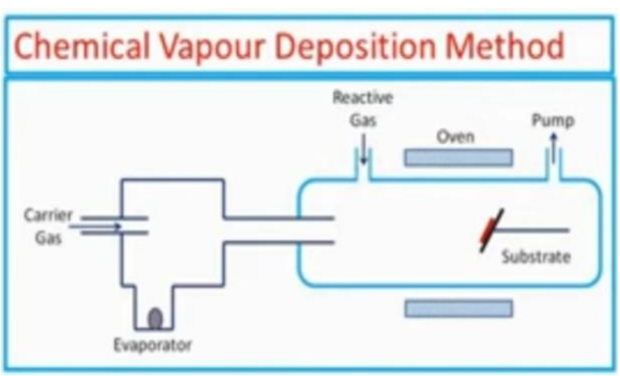


A spinning disc reactor (SDR) creates nanoparticles by spinning them together. It has a rotating disc inside of a chamber or reactor where the physical characteristics including temperature, can be regulated. Typically, nitrogen or other inert gases are used to fill the reactor. Remove oxygen from the inside to prevent chemical reaction . The disc rotates at various speeds where precursor and water are pumped in as the liquid. The spinning leads to the fusion of the atoms or molecules, together and are combined, precipitated, collected and dried [24]. Various operational parameters including the,

a. Liquid flow rate
b. Speed of rotation
c. Liquid-to-precursor ratio
d. Feed position
e. Disc surface, etc.

Determine the properties of spinning disc reactor(SDR)-derived nanoparticles.

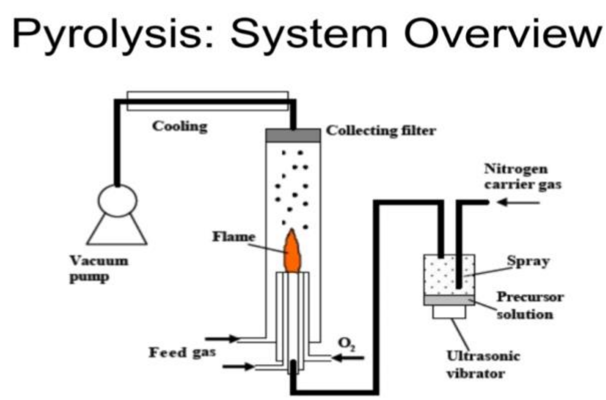
Chemical vapour deposition(CVD) :



Deposition of a thin film of gaseous reactants onto a substrate is known as chemical vapour deposition. Combining gas molecules causes the deposition to occur in a reaction chamber at room temperature. When a heated substrate contacts an acids, a chemical reaction takes places on the mixed gas surface . This reaction leaves a thin coating of the product on the substrate, restored and utilised surface. The affecting factor in CVD is the substrate temperature. The highly pure, homogenous, uniformity, hardness and strength are benefits of CVD.

Drawback-Special equipment is needed for CVD, and the gaseous by products are highly hazardous [25]

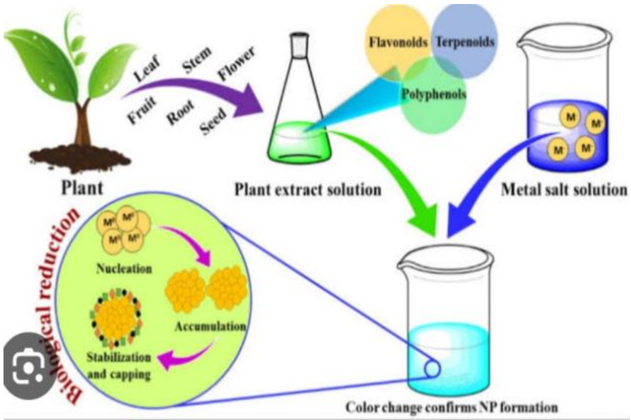
Pyrolysis:



The most widely utilised method for producing nanoparticles on big scale is pyrolysis. It involves using flame to burn a precursor. The precursor is either a liquid or a vapour that is injected under high pressure through a small hole into the furnace, where it burns [26]. The nanoparticles are then recovered by air-classifying by product gases. Some furnaces create high temperature for simple evaporation by using laser and plasma rather than flame [27].

Advantage-Simple, efficient, highly effective, continuous processes with high yield.

Biosynthesis:

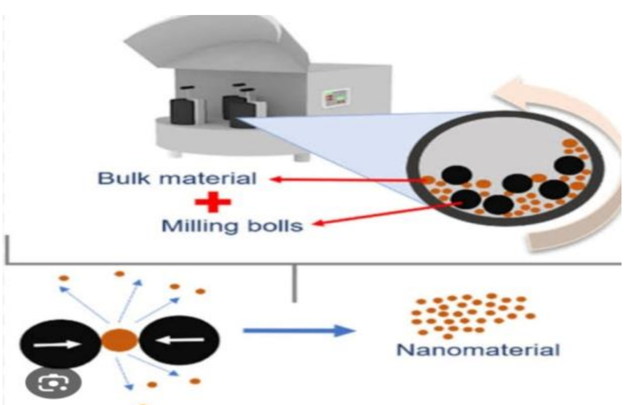


An ecofriendly method for creating non-toxic, biodegradable [28] nanoparticles is biosynthesised. Instead of using conventional chemicals, biosynthesis creates nanoparticles using precursors and elements like bacteria, plant extracts, fungi etc, and for bioreduction and capping applications. The biosynthesized nanoparticles have distinct and improved features that find use in biomedical applications [29].

Top down method:

A top down or destructive process involves breaking down a large material into tiny particles. Among them are mechanical milling, nanolithography, laser ablation, sputtering, and thermal breakdown are some one of the most used techniques for creating nanoparticles.

Mechanical Milling :

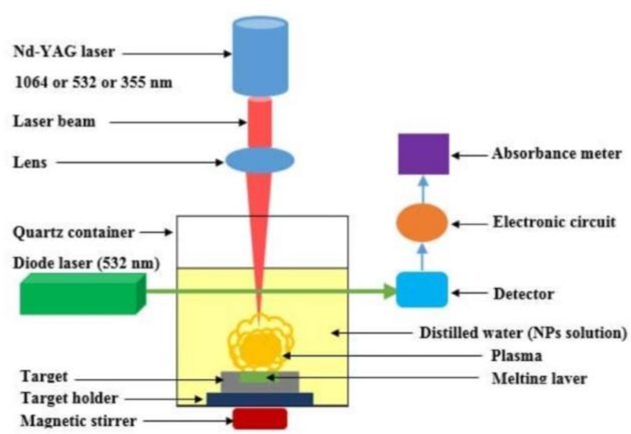


The most popular top down techniques for creating different nanoparticles is mechanical milling. When creating nanoparticles different elements are milled in an inert atmosphere using mechanical milling. Plastic deformation, which affects particle shape, is one of the influencing elements in mechanical milling [30]. Cold-welding increases particle size whereas fracture decreases it.

Nanolithography:

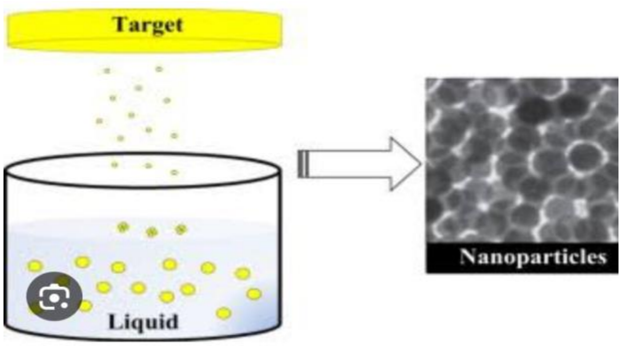
The study of creating structures with a minimum of one dimension between the sizes of 1 and 100nm is known as nanolithography. There are several methods for creating nanolithography including optical, electron-beam, multiphoton, nanoimprint, and scanning probe. Lithography [31], in general, refers to the process of printing a necessary shape or structure on a material that is light-sensitive that uses selective material removal to achieve the desired form and structure. Cost and the need for complicated equipment are drawbacks [32].

Laser ablation:



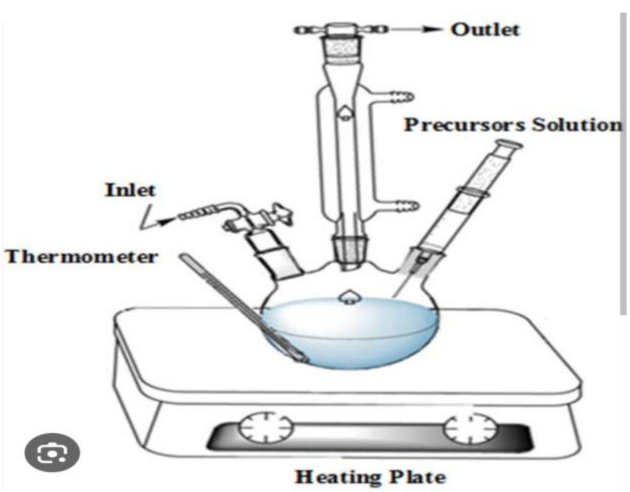
The procedure is known as laser ablation synthesis in solution (LASiS) is frequently used to create nanoparticles from a variety of solvents. The radiation of metal that is submerged in a liquid. A plasma plume is condensed by a laser beam, creating nanoparticles [33]. It is a reliable top-down approach that offers an alternative to the standard chemical reduction of metals to creating nanoparticles with a metal basis. LASiS offers a reliable method for creating nanoparticles in organic materials. It is a green method because it only uses solvents and water and doesn't need any chemicals or stabilizers.

Sputtering:



Sputtering is the deposition of nanoparticles on a surface by ejecting particles from it by interacting with ions. Sputtering typically involves depositing a thin coating of nanoparticles, followed by annealing. The substrate type, annealing temperature, and annealing time, among other factors [34].

Thermal decomposition:



Decomposition brought caused by heat. When heat is applied to a compound the chemical bonds are broken, causing thermal decomposition, an endothermic chemical reaction. Decomposition temperature is the precise temperature, at which an element begins to chemically breakdown. The particles are nanoscale formed when a chemical process takes place to breakdown the metal at a certain temperature.

Characterization of Nnaoparticles:

1. Particle Size:

In nanoparticle systems, particle size and size distribution are crucial factors. They determine the biological destiny, in vivo dispersion, toxicity, and targeting a nanoparticle system's capacity. Additionally, they can alter the stability, drug loading, and release of nanoparticle [35]. Photon-correlation spectroscopy or dynamic light scattering are now the quickest, and most frequent ways to estimate particle size. Photon-correlation spectroscopy needs the following : knowing the medium's viscosity and the particle's diameter is calculated by Brownian motion and scattering characteristics. The results obtained by photon-correlation spectroscopy are usually verified by scanning electron microscope (SEM) or transmission electron microscope (TEM) [36].

2. Particle Shape

Before being evaluated, the nanosuspension is cgaracterized by SEM and then lyophilized to produce solid particles. The solid particles have a platinum alloy coating [37].

3. **Zeta potential**:

Nanoparticle surface charge properties are frequently described using the zeta potential of the nanoparticle. It is reflective of a particle's electrical potential and impacted by the particle's composition and the medium which it is spread throughout. Nanoparticles with a zeta potential above (±) 30 mV are stable due to the surface charge's ability to prevent the accumulation of particles [38].

4. **Drug Entrapment Efficiency**:

By centrifugation at 10,000 rpm for 30 minutes at 50℃, the nanoparticles were removed from the aqueous medium. The resultant supernatant solution was then decanted and dissolved in phosphate buffered saline pH7.4. Thus the procedure was repeated twice to remove the unentrapped drug molecules completely. The amount of drug entrapped in the nanoparticles was determined as the difference between the total amount of drug used to prepare the nanoparticles and the amount of drug present in the aqueous medium.

Drug Entrapment Efficiency (%) = Amount of drug released from the lyophilised nanoparticles x 100 Amount of drug initially taken to prepare the Nanoparticles [39].

5. **Drug loading**:

A successful nanoparticulate system should ideally have a high drug-loading capacity, which will lower the amount of matrix materials needed, in order to administer. It is possible to load drugs by two techniques,

When incorporating production of nanoparticles (Incorporation technique).

The drug's absorption following the development of by exposing the carrier to a concentrated drug solution. (Adsorption / Absorption technique).

6. **X-ray diffraction (XRD) analysis**:

A common method for determining the morphology and structure of crystals is called x-ray diffraction. It is depending on the amount of constituent. This method is employed to establish the metallic nature of the particles provides information on size and shape of the unit cell as it relates to translational symmetry from peak positions and data on electron density, where the atoms are placed within the unit cell [40].

7. **Fourier Transform Infrared (FTIR) Spectroscopy**:

Determines the relationship between infrared and light wavelength to determine the type of linked functional groups and structural characteristics of biological extracts containing nanoparticles. The calculated spectra clearly show the well-known relationship between nanoparticle optical properties. The greenly synthesized silver nanoparticles by employing different leaf extracts was analysed using Fourier Transform Infrared Spectroscopy showed characteristics peak.

Conclusion:

Apprehension over the secondary effects related to the development of NPs and an increasing desire for greener technologies have arisen in the field of green and maintainable remediation. The acceptance of green synthesis promises not only to avoid secondary conservational contamination but also to reduce manufacturing costs. However, there are still gaps in the research that should be addressed to assist the development of the field. The synthesis of metal oxide NPs with microorganisms or plant extracts, using biological mechanisms, opens up tremendous opportunities to produce biocompatible and cost-effective particles with potential applications in the healthcare sector.

Synthesizing of NPs via the bio-green route has attracted a great deal of attention as it involves no harmful chemicals in its synthesis method. Hence, bio-green synthesized NPs could be promising materials, opening up new prospects in clinical, energy, and environmental research.

Ethical Approval

No ethical approval was necessary for this study.

Author Contribution

All authors made substantial contributions to the conception, design, acquisition, analysis, or interpretation of data for the work. They were involved in drafting the manuscript or revising it critically for important intellectual content. All authors gave final approval of the version to be published and agreed to be accountable for all aspects of the work, ensuring its accuracy and integrity.

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